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BIOMEDICAL TEST MATERIALS PROGRAM: FY 1991 UPDATE ON THE STORAGE STABILITY OF FISH OIL TEST MATERIALS

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U.S. DEPARTMENT OF COMMERCE
National Oceanic and Atmospheric Administration
National Marine Fisheries Service
Southeast Fisheries Science Center
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INTRODUCTION

This is the second in a series of updates on the storage stability trials carried out by the Quality Assurance Project of the Fish Oil Biomedical Test Materials Program (BTMP). The Fish Oil BTMP was established in 1986 by a Memorandum of Understanding between the National Marine Fisheries Service (NMFS) and the National Institutes of Health/Alcohol, Drug Abuse and Mental Health Administration (NIH/ADAMHA). The primary mission of this Program is to provide standard test materials to answer research questions regarding the mode of action of omega-3 (n-3) fatty acids in ameliorating heart disease, inflammatory diseases, and cancer, as well as their role in brain development. The role of the Charleston Laboratory of NMFS is to produce extensively analyzed and quality assured fish oil, n-3 ethyl ester concentrate of fish oils, and purified ethyl esters of eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA). These test materials are available to researchers approved by the Fish Oils Test Materials Advisory Committee (FOTMAC) of the NIH/ADAMHA.

Fish oils are susceptible to oxidation due to their high content of polyunsaturated fatty acids, especially EPA and DHA. Therefore, one responsibility of the Quality Assurance Project of the BTMP is to determine the stability of test materials under storage conditions which might be expected to be available in a biomedical research environment. This report summarizes the status of storage stability trials carried out through fiscal year 1991. A previous interim report was published in 1989 (Van Dolah et al., 1989b).

The process of lipid oxidation involves the initiation of an autocatalytic free radical process through the formation of an oxygen activated methylene group adjacent to a double bond of an unsaturated fatty acid (Nawar and Hultin, 1988). Peroxide formation can be catalyzed by light, low concentrations of metal ions (especially iron and copper), heat, and oxygen (Erickson and List, 1985). These peroxide intermediates can subsequently form aldehydes, ketones, alcohols, and other compounds which impart off-flavors to the product (Jackson, 1985; Chang, 1988). The degree of oxidation may be so low that chemical tests such as the peroxide value do not reflect a change, but sensory panels are able to detect certain off-flavors in products containing a peroxide value of less than 1 meq/kg (Frankel, 1980, 1985). These oxidation products, when present at levels as low as parts per million or billion, can make a product unacceptable (Coppen, 1990). Ultimately, polymerization of fatty acid chains via their peroxidized groups can occur. Cho et al. (1987) used oxygen absorption to compare the extent of oxidation of ethyl EPA and DHA with that of ethyl linolenate and linoleate. They found that both EPA and DHA were much more susceptible to oxidation and polymer formation than either of the other two shorter-chained ethyl esters. More importantly, deleterious health effects due to consumption of oxidized and polymerized oils have been reported (Piché et al., 1988; Gurr, 1990; Kubow, 1990). Thus, the use of highly unsaturated test materials which have not been properly handled during packaging or

storage may lead to as many confounding research results as the use of a material of undefined composition.

The purpose of the stability trials reported here was to determine stability under conditions similar to those present in biomedical research facilities. Therefore, in most studies a positive control (i.e., conditions under which massive product degradation would definitely occur) was not included. Many of these studies are ongoing and, therefore, some of the data presented are preliminary results of a multi-year effort. Information on later time points will be available through a final report summarizing the completed studies.

METHODS AND MATERIALS

Source of Test Materials

Vacuum deodorized fish oil (VDFO), n-3 ethyl ester concentrate of VDFO, purified EPA (approximately 95%), and purified DHA (approximately 90%) were produced at the Charleston Laboratory from a commercially produced, partially refined menhaden (*Brevoortia spp.*) fish oil (Zapata-Haynie Corp., Reedville, VA) using methods published in the Biomedical Test Materials Production Methods and Safety Manual (Joseph, 1989). Steam-deodorized fish oil derived from the same starting material was produced by Cherry-Burrell Corporation (Louisville, KY). Corn, olive, and safflower oils were purchased commercially and used as placebo oils. Tocopherols (alpha- and gamma-) and tertiary butylhydroquinone (TBHQ) antioxidant were added to placebo oils at the Charleston Laboratory so they would match levels added to the fish oil. Ethyl esters of commercially produced corn, olive, and safflower oils were also prepared at the Charleston Laboratory according to the procedures published by Joseph (1989).

Stability Assays

Assays utilized to assess stability of the test materials include fatty acid composition, peroxide value (PV), tocopherol content, and sensory analysis. Two additional assays utilized in the earliest studies were anisidine value and free fatty acid content. These assays were eliminated from later studies because they did not appear to be as sensitive to relatively large changes in product quality as some of the other tests, i.e., PV and fatty acid composition. All procedures used have been previously published (Van Dolah and Galloway, 1988). Fatty acid composition and tocopherol analyses were performed by capillary gas chromatography. Peroxide value was performed by the standard method for fats and oils, modified for autotitration (AOAC method # 965.33,

1990). Sensory analysis was performed by a trained sensory panel by whom samples were rated for specific odor and flavor characteristics on a 15 cm unstructured scale, where 0 represents "absent" and 15 represents "very strong". The sensory procedure used is a modification of the Quantitative Descriptive Analysis (QDA) method (Gooch and Galloway, 1988; Stone et al., 1974; Powers, 1984; Mecredy et al., 1974; Zook and Wessman, 1977). All analyses were performed in duplicate at each time point.

Bulk Packaged Materials

Stability studies were carried out on six test materials, each packaged in 100 ml bottles, with one bottle sampled at each time point. Storage temperatures and packaging materials varied, depending on the material stored. The design of each study is described below with the production lot number of the material identified in parentheses:

Study 1. Vacuum deodorized menhaden oil with antioxidants (L88216BB). Stability of bulk oil containing 1 mg/g each of α- and γ-tocopherol[1] and 0.02% TBHQ[2] was tested in two 100 ml container types, thick-walled polyethylene (Nalge Co., Rochester, NY) and thin-walled polyethylene (Consolidated Plastics Co., Twinsburg, OH), at 5°C. Stability of the product was also compared with that stored in thick-walled polyethylene at -40°C. Peroxide value was analyzed quarterly for 12 months, and then again at 18 months. Fatty acid composition, anisidine value, and tocopherols were analyzed at six month intervals. Sensory analysis was performed at 0 and 12 months storage.

Study 2. Vacuum deodorized menhaden oil without antioxidants (L88218BO). Stability of the bulk oil was tested in three 100 ml container types, thick-walled polyethylene, thin-walled polyethylene, and glass, at 5°C. Stability of the product was also compared with that stored in thick-walled polyethylene at -40°C. Peroxide value was analyzed quarterly for 12 months, and then again at 18 months. Fatty acid composition and anisidine value were analyzed at six month intervals. Sensory analysis was performed at 0 and 12 months storage.

Study 3. N-3 ethyl ester concentrate (L88168BF). Stability of bulk n-3 ethyl ester concentrate was tested on esters stored in 100 ml thick-walled polyethylene bottles at -40°C. Sampling for peroxide value, anisidine value, fatty acid composition, tocopherols, and sensory analysis was carried out quarterly for one year and annually thereafter.

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[1]Vitamin E-5-67 and Tenox GT-1, Eastman Chemical Products, Kingsport, TN

[2]Tenox 20-A, Eastman

Study 4. Corn oil ethyl ester (L89152VF). Stability of bulk corn oil ethyl esters was tested on esters stored in 100 ml thick-walled polyethylene bottles at -40°C. Quarterly sampling was carried out for 9 months to analyze for peroxide value, fatty acid composition, and tocopherols. Sensory analysis was performed at 0 and 9 months.

Study 5. EPA ethyl ester (L89180BI.4). EPA ethyl ester of 90% purity was stored in two different container types: nitrogen blanketed, vacuum sealed glass ampules and cryovac (Nalgene) vials stored at -40°C. Stability of the ester was determined at 0, 1, 3, 6, 12, and 24 months by peroxide value and fatty acid composition. Sensory analysis was performed at time 0 and 24 months.

Study 6. DHA ethyl ester (L89312BI.5). DHA ethyl ester of 93% purity was stored in cryovac (Nalgene) vials stored at -40°C. Stability of the ester was determined at 0, 3, 6, 9 and 12 months by peroxide value and fatty acid composition. Sensory analysis was performed at time 0 and at 12 months.

Soft-gelatin Encapsulated Materials

Steam- and vacuum-deodorized fish oil, n-3 ethyl ester concentrate, placebo vegetable oils, and ethyl esters of the placebo vegetable oils were encapsulated by a commercial encapsulator in soft gelatin capsules, containing 1 g of product per capsule, using the rotary die method described by Fair (1989). Stability trials were carried out on five different encapsulated materials.

Study 7. Encapsulated steam deodorized fish oil (L86339AA). A 12-month storage study entailing monthly sampling of encapsulated steam deodorized menhaden oil, packaged in 100 capsules/amber glass bottle and stored at 5°C, was completed early in FY89 and published (Van Dolah et al., 1989a). The study was extended to include quarterly sampling until 24 months, then yearly sampling for 3 or more years. The data presented in this report include quarterly timepoints through 24 months and annual timepoints thereafter for peroxide value, anisidine value, free fatty acids, fatty acid composition, moisture, tocopherols, and sensory analysis.

Study 8. Encapsulated vacuum deodorized fish oil (L88333BB). The encapsulated vacuum deodorized fish oil was stored 100 capsules/bottle in 100 ml thick-walled polyethylene bottles at 5°C. Samples were taken at 0, 12, 18, and 24 months to confirm comparability of the vacuum deodorized product with the steam deodorized product studied above. Samples were analyzed for peroxide value, anisidine value, moisture, fatty acid composition, tocopherols, and sensory analysis. This study is continuing through at least 36 months.

Study 9. Encapsulated n-3 ethyl ester concentrate (L88333BF). The encapsulated n-3 esters were stored 100 capsules/bottle in 100 ml thick-walled polyethylene bottles at 5°C.

Samples were taken quarterly for 1 year, then at six month intervals until 2 years, and analyzed for peroxide value, anisidine value, moisture, fatty acid composition, tocopherols, and sensory analysis. This study is continuing through at least 36 months.

Study 10. Encapsulated olive oil esters (L88333WF). The encapsulated esters were stored 100 capsules/bottle in 100 ml thick-walled polyethylene bottles at 5°C. Samples were taken at 0, 6, 12, 18 and 24 months and analyzed for peroxide value, anisidine value, moisture, fatty acid composition, tocopherols, and sensory analysis.

Study 11. Encapsulated corn oil esters (L89165VF). The encapsulated esters were stored 100 capsules/bottle in 100 ml thick-walled polyethylene bottles and stored at 5°C. Samples were taken quarterly for one year, and at six-month intervals thereafter, and analyzed for peroxide value, anisidine value, moisture, fatty acid composition, tocopherols, and sensory analysis.

RESULTS AND DISCUSSION

Bulk Packaged Materials

Studies 1 and 2 compared storage of VDMO at 5°C versus -40°C and at 5°C in three container types. Both with and without antioxidants, vacuum deodorized fish oil was stable through the eighteen month duration of the study when stored at -40°C, as assessed by peroxide value (Tables 1 and 3). At 5°C, glass bottles provided similar product stability as polyethylene bottles stored at -40°C. However, the oils stored in the polyethylene bottles at 5°C rapidly accumulated peroxides over the storage period, showing the effect of refrigerator temperature versus -40°C. Peroxide values of oils stored in the thick walled polyethylene were consistently lower than those in the thinner walled bottles, in direct correlation with their respective oxygen permeability. Anisidine value increased somewhat in the polyethylene bottles stored at 5°C, but did not change in those stored at -40°C or in glass. EPA and DHA content of any of the stored oils did not detectably decrease over a period of 18 months. The VDMO (containing 1 mg/g each of α and γ tocopherol and 0.02% TBHQ antioxidants) stored at 5°C in polyethylene bottles developed higher peroxide levels than those stored without antioxidants. Karahadian and Lindsay (1989b) found that adding 670 ppm of α-tocopherol to steam deodorized menhaden oil actually accelerated the rate of oxidation at 65°C, due to the prooxidant character of this antioxidant when present above a certain concentration. The primary sensory attributes in these products observed in our studies by the panel were mild "fishy", "grassy" and "beany" characteristics (Tables 2 and 4). Despite the dramatic differences in peroxide value at time 0 versus 18 months, no difference in product quality was detected by the sensory panel in terms of "total intensity" scores for odor and flavor. The PV measures primary products (hydroperoxides) of lipid oxidation, and does not give any information about

the secondary products (which are usually responsible for "off" flavors). Therefore, the PV may not correlate well with flavor (Jackson, 1985). However, "beany", "grassy" and "fishy" attributes did increase slightly over time, indicating that some aldehyde and ketone break-down products had formed. Stansby and Jellinek (1965) noted that the "green" flavor (resembling green grass or cucumber) intensified during early oxidation of menhaden oil, and that the "green" flavor was followed by fleeting "fishiness" before rancidity began to develop. Karahadian and Lindsay (1989a) were able to identify many of the odors and flavors in oxidized menhaden oils. They found that trans, cis-2,6-nonadienal was primarily responsible for the "green", "fresh fish" flavor in fish oil with trans-2-hexenal and 1,5-octadien-3-one adding heavier green components.

Study 3 determined stability of n-3 ethyl ester concentrates stored at -40°C in thick-walled polyethylene bottles (Table 5). Peroxide value rose over three years of storage from 5.8 to 10.9, with slight fluctuations between timepoints that may be due to variability between bottles. This change in peroxides did not result in an increase in anisidine value. There was no change in the EPA and DHA content of the esters over 36 months storage, nor in the α- and γ-tocopherol content. The ethyl esters of fish oil are characterized by sensory attributes which largely differ from those in the vacuum deodorized oil, particularly the "fruity/perfumy", "soapy/solvent" and "bitter" characteristics. The sensory panel observed no notable changes in flavor and odor of the ethyl esters, other than a slight decrease in "solvent" odor over time.

Study 4 determined the storage stability of corn oil ethyl esters in thick walled polyethylene bottles at -40°C (Table 6). This study was terminated after 9 months storage due to insufficient material. No change was observed in the peroxide value or fatty acid content of the product over the nine month period. Sensory attributes which were prominent in this product were "fruity/perfumy", "solvent", "raw green", and "liquor". The sensory panel did observe an increase in the "raw green" odor and flavor component during storage.

Study 5 determined the stability of purified EPA ethyl esters at -40°C in two different container types, glass ampules (Table 7) and polypropylene cryovials (Table 8). Both container types provided adequate storage stability with no degradation of EPA observed over 12 months storage. However, the glass ampules provided slightly better protection against oxidation as determined by a smaller change in peroxide value, with an initial value of 1.6 in both products and a 12 month value of 2.7 and 4.3, respectively, for glass versus polypropylene. An exception was observed between 12 and 24 months, when samples in glass ampules showed a much higher increase in PV over those in polypropylene vials. Similarly, "total intensity" scores for flavor and odor were higher for glass ampules versus polypropylene. Samples in both types of containers showed an increase in the sensory flavor attributes of "painty", "bitter", "acrid" and "soapy" over time.

Throughout this study, there was a small but constant increase found in the amount of EPA and total esters stored at -40°C in both types of containers. Thin layer chromatography (TLC) data have indicated that ethyl ester concentrates contain a small amount of monoacylglycerols, probably due to incomplete esterification. The apparent increase in purity of this product may be attributed to precipitation and/or adherence of these monoacylglycerols to the walls of the containers during frozen storage.

Study 6 determined storage stability of purified DHA ethyl esters in polypropylene vials at -40°C (Table 9). PV increased slightly over twelve months storage from 2.8 to 8.0. The sensory panel noted an increase in the "sweet" and "fruity/perfumy" attributes with a concomitant decrease in the "grassy," "fishy," and "cool/mint" flavors.

Gelatin Encapsulated Materials

Gelatin encapsulated steam deodorized fish oil (Study 7), previously reported to be stable over 12 months storage (Van Dolah et al., 1989a) continued to be stable through the 48 month sampling period (Table 10). These results are consistent with the observation of Ackman (1988) that no oxidation of PUFAs occurred in encapsulated fish oils stored at room temperature for approximately four years. DeKoning and Milkovitch (1989), on the other hand, observed considerable increases in PV of encapsulated fish oils stored at room temperature for one year. The primary sensory attributes of the encapsulated steam deodorized product included "fishy", "beany", "sweet" and "burnt". No change in "total intensity" scores were observed. However, the scores for the "burnt" and "sweet" characteristics appeared to increase until 18 months, and subsequently declined again. Karahadian and Lindsay (1989a) studied and identified volatile autoxidation compounds in fish oils. They observed that the decatrienals along with cis-4-heptenal caused distinct "burnt/fishy" flavors. Some of the flavors and aromas may be due to sources other than oxidation products. For example, Warner (1985) noted that "burnt" and residual "solvent" can result from processing steps or polymerized compounds remaining in improperly cleaned equipment. The standard operating procedures used in our pilot plant (Joseph, 1989) eliminate this as a probable cause.

Study 8 (Table 11) was initiated to compare the stability of encapsulated vacuum deodorized menhaden oil (VDMO) with the encapsulated steam deodorized menhaden oil (SDMO) shown in Table 10. No change in the encapsulated vacuum deodorized menhaden oil was observed over 24 months of storage at 5°C as assessed by peroxide value and EPA/DHA content. The sensory panel did note an increase in "total intensity of flavor" by the 24 month time point. Three sensory characteristics in particular increased during the study period, namely the "grassy", "fishy", and "burnt" odor attributes. The "burnt" odor attribute was absent at time 0. However, the "burnt" flavor did not intensify over time. Such increases upon storage suggest that these attributes

represent early oxidation products. It is worth noting that the encapsulated steam deodorized oil (Table 10) also contained these attributes and that they were present at time 0 and increased to higher levels than in the vacuum deodorized oil. The "burnt" characteristic has been observed only in encapsulated fish oil and never in bulk stored oil despite the higher peroxide values attained in the bulk stored oils. Hsieh et al., (1989) studied the odor components in winterized, undeodorized menhaden oil. These authors indicated that short-chain saturated and unsaturated aldehydes and ketones were responsible for the greasy, oily, oxidized oil and green grassy or green plant-like odors. Shukla and Perkins (1991) analyzed several encapsulated fish oils for the presence of oxidation products and found a wide range of peroxide values along with the presence of dimeric, trimeric, and oligomeric triacylglycerols. In addition to possible oxidation, the "burnt" characteristic could have resulted from non enzymatic browning of the gelatin capsule material during encapsulation.

Study 9 determined storage stability at 5°C of encapsulated n-3 ester concentrate (Table 12). Moisture content of the encapsulated ester concentrate increased with storage. However, the encapsulated esters demonstrated no significant change in EPA and DHA content or peroxide value over 24 months storage. Further, the sensory panel did not detect any change in quality. The primary attributes were similar to the bulk stored ethyl esters, "fishy", fruity/perfumy", and "soapy/solvent". Shukla and Perkins (1991) examined 6 retail encapsulated fish oil products (one of which was a fatty acid ethyl ester concentrate). The PV they obtained on this concentrate was approximately 21, or 10-fold higher than that in our samples. Ackman and Macpherson (1991) found that encapsulated fish oil free fatty acids showed PVs of less than 1 with an accompanying decline in anisidine values over time.

Soft gelatin encapsulated olive oil ethyl esters were tested at 5°C in Study 10 (Table 13). A slight increase in peroxide value and moisture content was observed during 24 months of storage. However, no change was observed in anisidine value or fatty acid composition. The sensory panel observed no dramatic change in odor or flavor characteristics during storage. Primary attributes of the olive oil esters were similar to those of the corn oil ethyl esters, namely "fruity/perfumy", "raw green", and a slight "cardboard" quality.

Stability of soft gelatin encapsulated corn oil ethyl esters was studied at 5°C (Study 11, Table 14). As with the other encapsulated products, no change in product quality was observed over 24 months storage in terms of fatty acid composition. Peroxide value increased in an apparent cyclic pattern, with a peak at 9 months, followed by a decline and then a further increase at 24 months storage. Part of this pattern may be due to variability between bottles. Moisture content also increased during storage. However, sensory analysis did not indicate a change in the product quality. The primary sensory attributes were similar to those observed in the bulk stored product, "fruity/perfumy", "raw green", "liquor", "soapy" and "solvent".

SUMMARY

Bulk VDMO stored in polyethylene at -40°C or in glass at 5°C remained stable in terms of peroxide value for 18 months. Stored in polyethylene at 5°C, VDMO formed substantial amounts of peroxides. None of the storage treatments resulted in any significant decrease in either EPA or DHA content. Encapsulation of VDMO or a similar product, SDMO, stabilized the oil in terms of both PV and n-3 content for at least three years at 5°C storage.

Both bulk packaged n-3 concentrate (-40°C) and encapsulated n-3 concentrate (5°C) were stable in terms of EPA and DHA content for at least 24 months. The bulk packaged esters demonstrated larger accumulations of peroxides than the oils at -40°C. However, encapsulation increased the storage stability, with no change in PV at 24 months storage.

All bulk and encapsulated placebo ethyl esters tested demonstrated equivalent storage stability as test materials. As with the fish oil products, the encapsulated materials demonstrated increased storage stability relative to bulk packaged materials.

Purified EPA and DHA ethyl esters were stable for at least 12 months at -40°C when stored in glass ampules or polypropylene cryovials. Polypropylene cryovials, however, may provide better protection for longer term storage.

All bulk packaged materials are adequately stable when stored at -40°C for at least two years. All encapsulated materials tested appear to be stable when stored at 5°C for two years and potentially longer.

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Table 1. Storage stability of bulk packed vacuum deodorized fish oil, containing antioxidants, stored at -40°C vs 5°C and in thick walled vs thin-walled polyethylene bottles (L88216BB).

TIME (months)	TREATMENT	PV	AV	a-Toc	g-Toc	EPA	DHA	TOT n-3
0		0.43	34.7	0.6	0.9	134	85	298
3	thick -40°C	0.44	-	-	-	-	-	-
	thick 5°C	2.75	-	-	-	-	-	-
	thin 5°C	3.06	-	-	-	-	-	-
6	thick -40°C	0.43	33.8	0.8	1.1	134	85	298
	thick 5°C	9.12	32.8	0.8	1.1	133	84	295
	thin 5°C	10.08	33.4	0.8	1.1	134	85	297
9	thick -40°C	0.41	-	-	-	-	-	-
	thick 5°C	11.43	-	-	-	-	-	-
	thin 5°C	15.71	-	-	-	-	-	-
12	thick -40°C	0.26	35.8	0.9	1.1	130	81	289
	thick 5°C	16.37	37.1	0.8	1.1	129	81	288
	thin 5°C	22.62	37.4	0.8	1.1	128	80	284
18	thick -40°C	0.49	35.0	0.8	1.1	133	84	296
	thick 5°C	21.61	37.3	0.8	1.0	131	82	292
	thin 5°C	34.26	37.8	0.8	1.0	130	81	289

Table 2. Sensory analysis, bulk oil with antioxidants, stored in three bottle types at 5°C or -40°C (L88216BB).

	Months Storage			
	0	12 MONTHS		
		-40°C THICK PE	5°C THICK PE	5°C THIN PE
SENSORY ANALYSIS:				
ODOR:				
TOTAL INTENSITY	2.9	2.9	2.1	2.3
BUTTERY	0	0	0	0
BEANY	0.3	0.1	0.3	0.4
RANCID	0	0	0	0
PAINTY	0	0	0	0
OXIDIZED	0	0	0	0
GRASSY	0.3	0.4	0.4	0.3
FISHY	0.3	1.5	0.9	0.9
BITTER	0	0	0	0
SWEET	0	0	0	0
FRUITY/PERFUMY	0.6	0	0	0
BURNT	0	0	0	0
DECOMPOSITION	0	0.4	0.2	0
LIQUOR	0	0	0	0.1
FLAVOR:				
TOTAL INTENSITY	3.0	3.3	2.2	2.1
BUTTERY	0	0	0	0
BEANY	0.3	0.3	0.1	0.4
RANCID	0	0	0	0
PAINTY	0.3	0	0	0
OXIDIZED	0	0	0	0
GRASSY	0.2	0.3	0.1	0.2
FISHY	0.9	1.8	0.8	1.3
BITTER	0	0	0	0
SWEET	0	0	0	0
FRUITY/PERFUMY	0.1	0	0	0
BURNT	0	0	0	0
DECOMPOSITION	0	0.2	0.4	0

Table 3. Bulk oil, without antioxidants, stored in three bottle types at 5°C or -40°C (L88218BO).

TIME (months)			PV	AV	EPA	DHA	TOT n-3
0			0.66	36.5	135	85	300
3	thick-walled PE	-40°C	0.98	-	-	-	-
	thick-walled PE	5°C	4.09	-	-	-	-
	thin-walled PE	5°C	8.53	-	-	-	-
	glass	5°C	1.35	-	-	-	-
6	thick-walled PE	-40°C	0.87	36.9	134	85	299
	thick-walled PE	5°C	7.86	35.8	132	84	295
	thin-walled PE	5°C	11.00	38.3	134	85	298
	glass	5°C	1.77	33.9	133	84	297
9	thick-walled PE	-40°C	0.97	-	-	-	-
	thick-walled PE	5°C	9.23	-	-	-	-
	thin-walled PE	5°C	11.82	-	-	-	-
	glass	5°C	0.84	-	-	-	-
12	thick-walled PE	-40°C	0.81	38.5	131	83	292
	thick-walled PE	5°C	11.83	41.6	130	82	289
	thin-walled PE	5°C	15.46	43.4	129	82	289
	glass	5°C	1.95	38.2	129	82	288
18	thick-walled PE	-40°C	1.46	37.3	135	85	300
	thick walled PE	5°C	16.63	42.2	133	84	296
	thin-walled PE	5°C	23.77	46.4	133	84	295
	glass	5°C	1.56	36.0	133	84	296

Table 4. Sensory analysis, bulk oil without antioxidants, stored in three bottle types at 5°C or -40°C (L88218BO).

	Months Storage				
	0	12 MONTHS			
		-40°C THICK PE	5°C THICK PE	5°C THIN PE	5°C GLASS
SENSORY ANALYSIS:					
ODOR:					
TOTAL INTENSITY	3.2	2.1	2.1	2.5	2.9
BUTTERY	0	0	0	0	0
BEANY	0.1	0.5	0.3	0.2	0.6
RANCID	0	0	0	0	0
PAINTY	0.9	0	0	0	0
OXIDIZED	0	0	0	0	0
GRASSY	0.1	0	0.5	0	0
FISHY	0.6	1.0	0	0.9	1.0
BITTER	0	0	0	0	0
SWEET	0	0	0	0	0
FRUITY/PERFUMY	0	0	0	0	0
BURNT	0	0	0	0	0
SOLVENT	0	0	0.4	0.3	0
FLAVOR:					
TOTAL INTENSITY	3.6	2.8	2.1	2.8	2.7
BUTTERY	0	0	0	0	0
BEANY	0.3	0.6	0.3	0.6	0.3
RANCID	0	0	0	0	0
PAINTY	1.3	0	0	0	0
OXIDIZED	0	0	0	0	0
GRASSY	0.1	0	0.7	0	0.5
FISHY	2.0	2.3	0.7	1.7	1.2
BITTER	0	0	0.2	0.4	0
SWEET	0	0	0	0	0
FRUITY/PERFUMY	0	0	0	0	0
BURNT	0	0	0	0	0
DECOMPOSITION	0	0	0	0.4	0

Table 5. Bulk n-3 ethyl esters stored at -40°C in thick walled polyethylene bottles (L88168BF).

	Months storage							
	0	3	6	9	12	18	24	36
EPA, mg/g	439	446	429	423	426	424	433	434
DHA, mg/g	240	243	232	226	228	228	231	232
TOTAL n-3, mg/g	783	789	765	761	766	760	776	775
a-TOCPOPHEROL, mg/g	0.9	0.9	0.9	0.9	0.9	0.8	0.9	0.9
g-TOCOPHEROL, mg/g	1.4	1.4	1.3	1.3	1.4	1.3	1.3	1.3
PEROXIDE VALUE, meq/kg	5.76	8.49	12.30	9.07	8.13	9.08	13.45	10.93
ANISIDINE VALUE	50.1	48.9	52.1	50.4	51.0	52.2	48.3	51.9
SENSORY ANALYSIS:								
ODOR:								
TOTAL INTENSITY	4.3	3.4	4.0	4.3	3.8	-	3.4	3.6
BUTTERY	0	0	0	0	0	-	0	0
BEANY	0	0	0	0	0	-	0	0.2
RANCID	0	0	0	0	0	-	0	0
PAINTY	0.9	1.5	0.9	0.5	0.7	-	0.6	0.1
OXIDIZED	0	0	0.1	0	0	-	0	0
GRASSY	0.1	0.2	0.4	0.2	0.2	-	0	0.2
FISHY	0	0	0.1	0	0	-	0.1	0
BITTER	0.7	0.4	0.7	0.2	0.6	-	0	0
SWEET	0	0	0	0	0	-	0	0
FRUITY/PERFUMY	1.4	1.1	0.7	1.8	1.1	-	1.3	0.9
BURNT	0	0	0	0	0	-	0	0
SOAPY	0.9	0.8	1.0	0.8	1.2	-	0	0
SOLVENT	1.3	1.1	1.4	1.4	1.8	-	0.4	0.6
FLAVOR:								
TOTAL INTENSITY	4.3	4.4	4.2	4.5	3.9	-	5.5	3.6
BUTTERY	0	0	0	0	0	-	0	0
BEANY	0.1	0.1	0	0.2	0	-	0	0
RANCID	0	0	0	0	0	-	0	0
PAINTY	0.4	1.1	1.0	0.7	0.8	-	0.4	0
OXIDIZED	0	0.2	0.1	0.1	0	-	0.2	0
GRASSY	0.3	0.1	1.0	0.6	0	-	0.5	0.1
FISHY	0.4	0.1	0.8	0.4	0	-	0.7	0.5
BITTER	1.3	2.0	1.0	0.8	2.0	-	2.6	0.9
SWEET	0	0	0	0	0	-	0	0
FRUITY/PERFUMY	0.9	0.7	0.2	1.2	1.5	-	0	1.0
BURNT	0	0	0	0	0	-	0	0
SOAPY	1.4	3.4	1.6	1.8	2.6	-	0.7	0.9
SOLVENT	1.0	0.8	1.2	2.0	2.3	-	1.8	0.9
ACRID	0	0	0	0	0	-	1.2	0.4
DECOMPOSITION/CPF*	0	0	0.5	0.3	0	-	0.5	0

* Characteristic protein flavor

Table 6. Bulk corn oil ethyl esters stored at -40°C in thick walled polyethylene bottles (L89152VF).

	Months storage			
	0	3	6	9
16:0, mg/g	100	99	104	105
18:1n-9, mg/g	223	223	231	234
18:2n-6, mg/g	492	493	501	507
total esters, mg/g	878	884	885	921
a-TOCPOPHEROL, mg/g	1.2	1.6	1.6	1.5
g-TOCOPHEROL, mg/g	2.0	2.2	2.2	2.1
PEROXIDE VALUE, meq/kg	3.07	4.23	3.26	3.30
SENSORY ANALYSIS:				
ODOR:				
TOTAL INTENSITY	3.7	-	-	3.9
BUTTERY	0	-	-	0
BEANY	0	-	-	0.3
RANCID	0	-	-	0
PAINTY	0.3	-	-	0.5
OXIDIZED	0	-	-	0
GRASSY	0.2	-	-	0
FISHY	0	-	-	0
BITTER	0	-	-	0
SWEET	0.3	-	-	0
FRUITY/PERFUMY	0.6	-	-	0.5
BURNT	0	-	-	0
SOLVENT	0.9	-	-	0.4
RAW GREEN	0.4	-	-	1.1
CARDBOARD	0	-	-	0
LIQUOR	0.6	-	-	0.7
FLAVOR:				
TOTAL INTENSITY	3.4	-	-	4.4
BUTTERY	0	-	-	0
BEANY	0	-	-	0.2
RANCID	0	-	-	0
PAINTY	0.5	-	-	0.3
OXIDIZED	0	-	-	0.1
GRASSY	0.3	-	-	0
FISHY	0	-	-	0
BITTER	0	-	-	0
SWEET	0.3	-	-	0
FRUITY/PERFUMY	0.7	-	-	0.5
BURNT	0	-	-	0
SOLVENT	0.8	-	-	0.5
SOAPY	0.6	-	-	0.3
RAW GREEN	1.4	-	-	2.1
ALMOND	0.3	-	-	0
LIQUOR	0.6	-	-	0.5
CARDBOARD	0.8	-	-	0

Table 7. Purified EPA ethyl ester stored at -40°C in glass ampules (L89180BI.4).

	Months storage					
	0	1	3	6	12	24
EPA, mg/g	902	904	908	916	945	957
total esters, mg/g	928	931	935	944	968	979
a-TOCPOPHEROL, mg/g	<0.1	-	-	-	-	<0.1
g-TOCOPHEROL, mg/g	1.4	-	-	-	-	1.3
PEROXIDE VALUE, meq/kg	1.61	1.68	1.38	1.28	2.68	39.10
SENSORY ANALYSIS:						
ODOR:						
TOTAL INTENSITY	2.7	-	-	-	-	4.2
BUTTERY	0	-	-	-	-	0
BEANY	0.1	-	-	-	-	0
RANCID	0	-	-	-	-	0
PAINTY	0.2	-	-	-	-	1.2
OXIDIZED	0	-	-	-	-	0
GRASSY	0.7	-	-	-	-	0
FISHY	0.2	-	-	-	-	0
BITTER	0	-	-	-	-	0
SWEET	0.1	-	-	-	-	0
FRUITY/PERFUMY	0.7	-	-	-	-	0.6
BURNT	0	-	-	-	-	0
SOLVENT	0.7	-	-	-	-	1.0
LIQUOR	0.7	-	-	-	-	1.6
RAW GREEN	0	-	-	-	-	0.4
FLAVOR:						
TOTAL INTENSITY	3.0	-	-	-	-	5.5
BUTTERY	0	-	-	-	-	0
BEANY	0	-	-	-	-	1.7
RANCID	0.3	-	-	-	-	0
PAINTY	0	-	-	-	-	2.2
OXIDIZED	0	-	-	-	-	1.7
GRASSY	0.9	-	-	-	-	0.5
FISHY	0.8	-	-	-	-	0
BITTER	0.2	-	-	-	-	3.0
SWEET	0.6	-	-	-	-	0
FRUITY/PERFUMY	1.1	-	-	-	-	0
BURNT	0	-	-	-	-	0
SOLVENT	0.5	-	-	-	-	1.8
ACRID	0	-	-	-	-	1.7
LIQUOR	0	-	-	-	-	0

Table 8. Purified EPA ethyl ester stored at -40°C in polypropylene cryovials (L89180BI.4).

	Months storage					
	0	1	3	6	12	24
EPA, mg/g	902	898	908	922	944	969
total esters, mg/g	928	929	934	950	966	988
a-TOCPOPHEROL, mg/g	<0.1	-	-	-	-	<0.1
g-TOCOPHEROL, mg/g	1.4	-	-	-	-	1.3
PEROXIDE VALUE, meq/kg	1.61	1.68	1.82	2.62	4.28	14.81
SENSORY ANALYSIS:						
ODOR:						
TOTAL INTENSITY	2.7	-	-	-	-	3.4
BUTTERY	0	-	-	-	-	0
BEANY	0.1	-	-	-	-	0.4
RANCID	0	-	-	-	-	0
PAINTY	0.2	-	-	-	-	1.0
OXIDIZED	0	-	-	-	-	0
GRASSY	0.7	-	-	-	-	0
FISHY	0.2	-	-	-	-	0
BITTER	0	-	-	-	-	0
SWEET	0.1	-	-	-	-	0.5
FRUITY/PERFUMY	0.7	-	-	-	-	0.9
BURNT	0	-	-	-	-	0.2
SOLVENT	0.7	-	-	-	-	1.0
ALMOND	0.7	-	-	-	-	0
YEAST	0	-	-	-	-	0.3
FLAVOR:						
TOTAL INTENSITY	3.0	-	-	-	-	4.3
BUTTERY	0	-	-	-	-	0
BEANY	0	-	-	-	-	0.2
RANCID	0.3	-	-	-	-	0
PAINTY	0	-	-	-	-	2.0
OXIDIZED	0	-	-	-	-	0.4
GRASSY	0.9	-	-	-	-	0.3
FISHY	0.8	-	-	-	-	0.2
BITTER	0.2	-	-	-	-	1.7
SWEET	0.6	-	-	-	-	0.1
FRUITY/PERFUMY	1.1	-	-	-	-	0.4
BURNT	0	-	-	-	-	0
SOLVENT	0.5	-	-	-	-	1.0
ACRID	0	-	-	-	-	2.2
SOAPY	0	-	-	-	-	2.1
LIQUOR	0	-	-	-	-	1.2
RAW GREEN	0	-	-	-	-	0.4

Table 9. Purified DHA ethyl ester stored at -40°C in polypropylene cryovials (L89312BI.5).

	Months storage				
	0	3	6	9	12
DHA, mg/g	933	909	921	949	925
total esters, mg/g	974	950	956	990	955
a-TOCPOPHEROL, mg/g	0.6	-	-	-	-
g-TOCOPHEROL, mg/g	0.1	-	-	-	-
PEROXIDE VALUE, meq/kg	2.83	-	5.29	3.84	8.00
CHOLESTEROL, mg/g	0.1	-	-	-	-
MOISTURE, ug/g	288.0	-	-	-	-
SENSORY ANALYSIS:					
ODOR:					
TOTAL INTENSITY	2.8	-	-	-	4.1
BUTTERY	0	-	-	-	0
BEANY	0.1	-	-	-	0.2
RANCID	0	-	-	-	0
PAINTY	0.3	-	-	-	0.3
OXIDIZED	0.3	-	-	-	0
GRASSY	0.5	-	-	-	0
FISHY	0.5	-	-	-	0
BITTER	0	-	-	-	0
SWEET	0	-	-	-	0.9
FRUITY/PERFUMY	0	-	-	-	1.3
BURNT	0.3	-	-	-	0
SOLVENT	0.5	-	-	-	0.5
SOAPY	0.2	-	-	-	0.1
APPLE	0	-	-	-	0.1
RAW GREEN	0	-	-	-	0.2
LIQUOR	0	-	-	-	0.1
ACRID	0	-	-	-	0.1
FLAVOR:					
TOTAL INTENSITY	5.1	-	-	-	4.7
BUTTERY	0	-	-	-	0
BEANY	0	-	-	-	0
RANCID	0	-	-	-	0
PAINTY	1.2	-	-	-	0.4
OXIDIZED	0	-	-	-	0
GRASSY	0.8	-	-	-	0
FISHY	1.8	-	-	-	0.7
BITTER	1.2	-	-	-	1.4
SWEET	0	-	-	-	0.5
FRUITY/PERFUMY	0.4	-	-	-	0.6
BURNT	0	-	-	-	0
SOLVENT	1.0	-	-	-	1.7
SOAPY	0.3	-	-	-	1.4
APPLE	0	-	-	-	0.1
LIQUOR	0	-	-	-	0.1
RAW GREEN	0	-	-	-	0.2
CPF	0.2	-	-	-	0
COOL/MINT	0.8	-	-	-	0
ACRID	0.1	-	-	-	0.1
MUSTY	0.3	-	-	-	0.4
CARDBOARD	0	-	-	-	0.2

Table 10 . Storage stability of encapsulated steam-deodorized menhaden oil at 5°C (A86339A).

MONTHS IN STORAGE	0	3	6	9	12	15	18	21	24	36	48
EPA, mg/g	118	115	120	122	120	-	118	118	119	115	125
DHA, mg/g	68	70	71	74	73	-	73	72	73	70	76
TOTAL n-3, mg/g	249	255	260	268	250	-	259	263	263	254	276
FREE FATTY ACIDS, %	-	-	0.12	-	0.02	-	0.13	0.04	0.14	0.04	0.08
PEROXIDE VALUE	1.40	2.75	1.53	1.49	1.49	1.78	1.89	2.17	1.25	2.16	1.45
IODINE VALUE	177	178	-	-	-	-	172	-	-	-	-
ANISIDINE VALUE	18.2	-	-	19.5	17.9	19.1	-	17.7	19.0	19.9	19.3
ANTIOXIDANT CONTENT:											
a-TOCOPHEROL, mg/g	1.0	1.2	1.0	-	-	-	1.0	1.0	-	0.9	1.0
TBHQ, mg/g	-	0.12	0.12	-	0.10	-	-	-	-	-	-
MOISTURE, ug/g	200	215	287	202	202	-	279	270	244	295	328
SENSORY ANALYSIS:											
ODOR:											
TOTAL INTENSITY	3.6	3.0	3.5	4.1	3.8	4.0	4.1	3.4	4.0	3.5	4.9
BUTTERY	0	0	0	0	0	0	0	0	0	0	0
BEANY	0.1	0.1	0.1	0	0.1	0.1	0.2	0.2	0.3	0.5	0.7
RANCID	0.1	0	0.1	0	0	0	0	0	0	0	0
PAINTY	0.2	0	0	0	0.1	0	0	0	0.2	0	0
OXIDIZED	0.3	0.3	0.1	0.2	0	0	0	0.1	0	0.4	0.1
GRASSY	0.1	0	0	0.1	0	0	0	0	0	0	0
FISHY	1.0	0.3	0.3	0.4	0.8	0.1	0.1	0.3	0.4	0.9	1.8
BITTER	0	0	0	0	0	0	0	0	0	0	0
SWEET	0.8	0.8	1.4	0.8	0.5	1.4	1.3	1.4	1.6	0.2	0.5
FRUITY/PERFUMY	0.2	0	0	0	0	0	0	0	0	0	0
BURNT	1.3	1.6	1.8	1.9	1.4	1.8	2.4	2.4	2.2	2.3	2.0
DECOMPOSITION	0	0	0	0	0	0	0	0	0	0	0.5
FLAVOR:											
TOTAL INTENSITY	3.8	3.3	3.4	4.7	4.2	4.3	5.1	3.1	4.2	4.1	4.9
BUTTERY	0.3	0.1	0.1	0.5	0	0	0	0	0	0	0
BEANY	0.3	0.2	0.1	0	0.1	0	0.1	0.2	0.2	0.7	0.8
RANCID	0.2	0	0	0	0	0	0.4	0	0	0	0
PAINTY	0.1	0	0.1	0.1	0.5	0	0	0	0.4	0.4	0.3
OXIDIZED	0.2	0.3	0.2	0.4	0	0	0	0	0.3	0.5	0
GRASSY	0.2	0.1	0.1	0	0	0	0	0	0.1	0.2	0.2
FISHY	2.1	1.4	0.5	1.3	2.3	0.3	0.6	0.3	1.2	1.5	2.4
BITTER	0.1	0	0.3	0.6	0	0	0	0	0	0	0
SWEET	0.7	0.4	1.2	0.5	0.5	1.2	1.4	0.8	1.4	0	0.8
FRUITY/PERFUMY	0.1	0	0.1	0.2	0	0	0	0	0	0	0
BURNT	0.9	1.7	2.0	1.4	2.0	3.3	3.0	2.4	2.7	1.5	1.8
DECOMPOSITION	0	0	0	0	0	0	0.8	0	0	0	0.4

Table 11. Storage stability of encapsulated vacuum-deodorized menhaden oil at 5 C (L88333BB).

	Months Storage			
WEEKS IN STORAGE	0	12	18	24
EPA, mg/g	136	140	140	139
DHA, mg/g	81	84	84	84
TOTAL n-3, mg/g	308	305	304	304
PEROXIDE VALUE, meq/kg	1.22	0.97	1.08	1.31
ANISIDINE VALUE	37.0	-	31.6	29.5
MOISTURE, ug/g	302	-	-	454
ANTIOXIDANT CONTENT:				
a-TOCOPHEROL, mg/g	0.9	0.9	1.0	0.9
g-TOCOPHEROL, mg/g	1.0	1.0	1.0	0.9
SENSORY ANALYSIS:				
ODOR:				
TOTAL INTENSITY	3.8	4.1	4.0	4.4
BUTTERY	0	0	0	0
BEANY	0.8	0	0.3	0.4
RANCID	0.2	0	0	0
PAINTY	0.3	0.8	0.3	0.5
OXIDIZED	0	0.4	0.3	0.2
GRASSY	0	0	0	0.4
FISHY	0.8	2.1	2.0	1.9
BITTER	0	0	0	0
SWEET	0.2	0	0	0
FRUITY/PERFUMY	0	0	0	0.1
BURNT	0.5	0.2	0.5	1.1
DECOMPOSITION	0	0.2	0	0.4
FLAVOR:				
TOTAL INTENSITY	4.2	4.9	4.8	5.2
BUTTERY	0	0	0	0
BEANY	0.7	0.6	0.1	0.1
RANCID	0.1	0	0	0
PAINTY	0.3	0	0.5	0.8
OXIDIZED	0	0.9	0	0
GRASSY	0.2	0	0.1	0
FISHY	0	3.5	3.4	3.3
BITTER	0	0	0	0
SWEET	0.2	0.3	0	0
FRUITY/PERFUMY	0	0	0	0
BURNT	0.6	0	0	0.2
DECOMPOSITION/CPF*	0.3	1.6	0.6	1.6
RAW GREEN	0	0.5	0	0

*Characteristic protein flavor

Table 12. Storage Stability of Soft Gelatin Encapsulated n-3 Ethyl Esters Stored at 5°C (L88333BF).

	Months Storage						
	0	3	6	9	12	18	24
EPA, mg/g	414	408	402	-	405	425	414
DHA, mg/g	236	229	227	-	229	239	235
TOTAL n-3, mg/g	788	758	748	-	750	786	761
a-TOCPOPHEROL, mg/g	1.0	1.0	0.8	-	1.0	1.0	0.9
g-TOCOPHEROL, mg/g	1.7	1.7	1.6	-	1.0	1.6	1.7
PEROXIDE VALUE, meq/kg	1.92	1.82	1.79	2.00	1.97	2.10	2.24
ANISIDINE VALUE	39.6	37.4	38.5	-	37.8	34.7	33.4
MOISTURE, ug/g	465	586	481	-	585	-	750
SENSORY ANALYSIS:							
ODOR:							
TOTAL INTENSITY	5.0	-	3.7	5.4	4.5	3.6	4.9
BUTTERY	0	-	0	0	0	0	0
BEANY	0	-	0.1	0	0	0.3	0
RANCID	0	-	0	0	0	0	0
PAINTY	1.1	-	0	1.2	1.2	0.3	0.7
OXIDIZED	0	-	0	0.6	0	0	0
GRASSY	0.4	-	0	0	0.1	0.1	1.1
FISHY	0	-	0	0.5	0	0.5	0
BITTER	0.9	-	0.7	1.0	0	0	0
SWEET	0	-	0	0	0.2	0	0.2
FRUITY/PERFUMY	1.5	-	1.8	1.5	1.3	0.9	2.6
BURNT	0	-	0	0	0	0	0
LIQUOR	0	-	0	0	0	0	0.2
SOAPY	1.6	-	0	1.6	1.5	0.3	0.6
SOLVENT	1.5	-	1.9	2.6	0.9	1.0	0.9
ACRID	0	-	0	0	0.5	0	0.3
FLAVOR:							
TOTAL INTENSITY	5.3	-	4.2	5.8	6.0	5.4	4.9
BUTTERY	0	-	0	0	0	0	0
BEANY	0	-	0	0	0	0	0
RANCID	0	-	0.5	0	0	0	0
PAINTY	1.4	-	0	1.2	1.3	0	0.2
OXIDIZED	0.2	-	0	0.3	0	0	0
GRASSY	0.4	-	0	0	0.2	0	0.1
FISHY	0.3	-	0.2	1.5	0.3	2.3	0.1
BITTER	2.0	-	1.4	1.0	1.1	1.0	0.3
SWEET	0	-	0	0	0	0	0
FRUITY/PERFUMY	1.3	-	1.5	0.4	1.4	0.6	2.0
BURNT	0	-	0	0	0	0	0
CARDBOARD	0	-	0	0.7	0	0.2	0.4
ACRID	0	-	0	0	1.3	0.4	0.8
CPF*	0	-	0	0	0.2	0	0
RAW GREEN	0	-	0	0	0.4	0	0.1
LIQUOR	0	-	0	0	0	0	0.2
SOAPY	2.9	-	0	3.3	2.4	0.9	0.5
SOLVENT	2.4	-	1.5	3.0	1.8	1.1	1.1

*Characteristic protein flavor

Table 13. Storage Stability of Soft Gelatin Encapsulated Ethyl Esters of Olive Oil Stored at 5°C (L88333WF).

	Months Storage				
	0	6	12	18	24
16:0, mg/g	102	105	103	110	110
18:1n-9, mg/g	605	610	625	641	644
18:2n-6, mg/g	94	95	97	98	98
a-TOCPOPHEROL, mg/g	1.1	0.9	0.9	0.9	1.0
g-TOCOPHEROL, mg/g	1.0	0.9	0.9	0.8	0.9
PEROXIDE VALUE, meq/kg	2.34	2.60	3.30	3.27	5.06
ANISIDINE VALUE	4.8	5.7	5.9	4.7	5.0
MOISTURE, ug/g	422	416	445	-	667
SENSORY ANALYSIS:					
ODOR:					
TOTAL INTENSITY	5.1	4.0	4.9	4.3	4.8
BUTTERY	0	0	0	0	0.1
BEANY	0.2	0	0.1	0	0
RANCID	0.1	0	0.2	0	0
PAINTY	0.9	0.1	0.2	0.1	0.2
OXIDIZED	0.1	0	0	0	0
GRASSY	0.5	0.1	0	0	0
FISHY	0	0	0	0	0
BITTER	0.2	0	0	0	0
SWEET	0.3	0	0.5	0	0.8
FRUITY/PERFUMY	0.7	0	0.9	0.6	2.3
BURNT	0	0	0	0	0
SOLVENT	0.5	0	0.2	0.4	0.1
RAW GREEN	0.5	1.5	2.8	1.7	0.2
LIQUOR	1.7	1.9	1.4	0.6	0.7
CARDBOARD	0.4	1.2	0	0.1	1.1
SOAPY	0.3	0	0	0.2	0
WASSAIL	0	0	0	0	1.0
YEAST	0.3	0	0	0	0.3
ACRID	0	0	0.2	0	0.3
FLAVOR:					
TOTAL INTENSITY	5.0	4.5	5.4	4.0	4.3
BUTTERY	0	0	0	0.2	0
BEANY	0	0	0	0	0
RANCID	0.8	0.3	0	0	0
PAINTY	1.1	0.5	0.2	0.4	0.1
OXIDIZED	0	0	0.3	0.1	0.1
GRASSY	0.6	0	0.3	0	0
FISHY	0	0	0	0	0
BITTER	0.3	1.4	0	0.3	0
SWEET	0.5	0	0	0	0.4
FRUITY/PERFUMY	0.6	0	0.7	0.4	1.2
BURNT	0	0	0	0	0
SOLVENT	0.7	0.8	0.2	0.3	0.5
CARDBOARD	0.4	1.7	0	0.4	1.1
SOAPY	1.2	0	0	0.6	0.1
MUSTY	0	0	0	0.2	0.5
YEAST	0	0	0	0	0.2
WASSAIL	0	0	0	0	0.5
RAW GREEN	0.4	1.8	3.1	2.3	0.6
LIQUOR	1.2	1.9	1.0	0	0.8

Table 14. Storage Stability of Soft Gelatin Encapsulated Ethyl Esters of Corn Oil Stored at 5°C (L89165VF).

	Months Storage						
	0	3	6	9	12	18	24
16:0, mg/g	103	97	105	106	110	99	104
18:1n-9, mg/g	229	225	232	235	243	222	235
18:2n-6, mg/g	501	497	513	512	520	480	514
a-TOCPOPHEROL, mg/g	1.5	-	1.6	1.4	1.7	1.6	1.6
g-TOCOPHEROL, mg/g	2.1	-	2.2	2.0	2.2	2.2	2.2
PEROXIDE VALUE, meq/kg	3.17	5.76	5.45	6.33	5.90	3.29	8.60
ANISIDINE VALUE	6.1	-	5.9	-	3.4	5.9	-
MOISTURE, ug/g	502	-	492	-	-	-	-
SENSORY ANALYSIS:							
ODOR:							
TOTAL INTENSITY	3.5	-	4.2	4.1	3.7	3.4	4.1
BUTTERY	0	-	0	0	0.1	0	0
BEANY	0	-	0.3	0.6	0	0	0
RANCID	0	-	0	0	0	0	0
PAINTY	0.1	-	0.3	0.1	0.3	0.5	0.2
OXIDIZED	0	-	0	0.2	0	0	0.1
GRASSY	0	-	0	0	0	0.2	0
FISHY	0	-	0	0	0	0	0
BITTER	0.1	-	0	0	0	0	0
SWEET	0.4	-	0.3	0.3	0	0	0
FRUITY/PERFUMY	0	-	0.5	0.4	1.1	1.1	0.3
BURNT	0	-	0.1	0.5	0	0	0
SOAPY	0	-	0	0	0		0.3
SOLVENT	0	-	0.6	0.3	0.1	0.7	0.7
RAW GREEN	1.5	-	1.8	1.2	0.7	1.0	1.7
CARDBOARD	0.8	-	0	0	0.2	0	0
LIQUOR	1.6	-	1.2	0.8	0.4	0	0.9
FLAVOR:							
TOTAL INTENSITY	3.9	-	3.7	4.3	3.8	3.3	4.4
BUTTERY	0	-	0	0	0.2	0	0
BEANY	0	-	0.3	0.3	0	0	0.1
RANCID	0.2	-	0	0	0	0	0
PAINTY	0	-	0	0.2	0.3	0.2	0.2
OXIDIZED	0	-	0.5	0.8	0	0	0
GRASSY	0	-	0	0.2	0	0.5	0
FISHY	0	-	0.4	0	0	0	0
BITTER	0.3	-	0	0	0	0.2	0.1
SWEET	0.4	-	0.3	0.2	0	0	0.2
FRUITY/PERFUMY	0	-	0.9	0.3	0.7	0.8	0.3
BURNT	0	-	0.2	0	0	0	0
SOAPY	0	-	0	0.6	0.1	0.7	0.9
SOLVENT	0	-	0.4	0.5	0.2	0.4	0.6
RAW GREEN	2.0	-	3.1	1.5	0.8	1.4	2.5
ACRID	0	-	0.4	0.1	0	0	0.1
LIQUOR	1.7	-	0.6	0.8	0.1	0	0.3
MUSTY	0	-	0	0	0	0.3	0.4
CARDBOARD	0.4	-	0	0	0.5	0	0